Health Consultation

Exposure Investigation Workplan

Lower Elwha Klallam Tribe Shellfish Beds Near Port Angeles Harbor Port Angeles, Clallam County, Washington

December 13, 2002

Prepared by

The Washington State Department of Health Under a Cooperative Agreement with the Agency for Toxic Substances and Disease Registry



Foreword

The Washington State Department of Health (DOH) has prepared this health consultation in cooperation with the Agency for Toxic Substances and Disease Registry (ATSDR) and the Lower Elwha Klallam Tribe (Tribe). ATSDR is part of the U.S. Department of Health and Human Services and is the principal federal public health agency responsible for health issues related to hazardous waste. This health consultation was prepared in accordance with methodologies and guidelines developed by ATSDR.

The purpose of this health consultation is to identify and prevent harmful human health effects resulting from exposure to hazardous substances in the environment. Health consultations focus on specific health issues so that DOH can respond quickly to requests from concerned residents or agencies for health information on hazardous substances. DOH evaluates sampling data collected from a hazardous waste site, determines whether exposures have occurred or could occur, reports any potential harmful effects, and recommends actions to protect public health. The findings in this report are relevant to conditions at the site during the time of this health consultation, and should not necessarily be relied upon if site conditions or land use changes in the future.

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For more information about ATSDR, contact the ATSDR Information Center at 1-888-422-8737 or visit the agency's Web site: www.atsdr.cdc.gov/.

Glossary

| Agency for Toxic |
|-------------------------|
| Substances and |
| Disease Registry |
| (ATSDR) |

The principal federal public health agency involved with hazardous waste issues, responsible for preventing or reducing the harmful effects of exposure to hazardous substances on human health and quality of life. ATSDR is part of the U.S. Department of Health and Human Services.

Carcinogen

Any substance that can cause or contribute to the production of cancer.

Comparison value

A concentration of a chemical in soil, air or water that, if exceeded, requires further evaluation as a contaminant of potential health concern. The terms comparison value and screening level are often used synonymously.

Contaminant

Any chemical that exists in the environment or living organisms that is not normally found there.

Exposure

Contact with a chemical by swallowing, by breathing, or by direct contact (such as through the skin or eyes). Exposure may be short-term (acute) or long-term (chronic).

Hazardous substance

Any material that poses a threat to public health and/or the environment. Typical hazardous substances are materials that are toxic, corrosive, ignitable, explosive, or chemically reactive.

Media

Soil, water, air, plants, animals, or any other part of the environment that can contain contaminants.

Organic

Compounds composed of carbon, including materials such as solvents, oils, and pesticides which are not easily dissolved in water.

| Parts per trillion |
|--------------------|
| (ppt)/Parts per |
| million (ppm) |

Units commonly used to express low concentrations of contaminants. For example, 1 ounce of trichloroethylene (TCE) in 1 million ounces of water is 1 ppm. 1 ounce of TCE in 1 trillion ounces of water is 1 ppt.

Remedial investigation

A study designed to collect the data necessary to determine the nature and extent of contamination at a site.

Volatile organic compound (VOC)

An organic (carbon-containing) compound that evaporates (volatilizes) easily at room temperature. A significant number of the VOCs are commonly used as solvents.

Background and Statement of Issues

Purpose of Exposure Investigation

The Lower Elwha Klallam Tribe requested that the Washington State Department of Health (DOH) evaluate whether their subsistence-level consumption of shellfish, collected in the vicinity of the Port Angeles Harbor, poses a health threat. Historically, the Harbor received effluent from two paper mills, including the former Rayonier Pulp Mill, which is located closest to the areas used by tribal members for shellfish collection, a plywood mill, and the city of Port Angeles. The effluent from the Rayonier mill is a known source of dioxins and furans. No information is available to indicate dioxins and furans are associated with the other sources; however, they may be potential sources. Dioxins and furans are the primary chemicals of concern to the Tribe. The Agency for Toxic Substances and Disease Registry (ATSDR) is providing a portion of the funding to conduct this exposure investigation (EI).

Background

The above-named historic effluent sources within the harbor are located within the Port Angeles city limits, in Clallam County, Washington. Port Angeles Harbor, which is formed by the extension of Ediz Hook into the Straight, is located immediately north of the city (Figure 1). The marine waters between the harbor and Dungeness Spit, located to the east, are considered an important subsistence fishery resource for the Tribe.

One of the effluent sources, the Rayonier pulp mill, which operated from 1929 to 1997, used an ammonia-based acid sulfite process for the production of various grades of pulp. Chlorine and chlorine compounds, that contribute to the production of dioxins and furans, were used in the bleaching process. Dioxins and furans were also released in effluent from other sources at the mill, such as the hog -fuel ash that was removed by the wet direct-contact air emission scrubbers.

Prior to the installation of the primary treatment plant at the mill in 1972, effluent (solid and liquid) generated during the pulp-making process, and surface water runoff, were discharged directly along the Port Angeles Harbor shoreline through a number of individual outfalls located adjacent to the mill. Data collected by Rayonier in 1963 indicate that effluent generally moved east from the mill site. Rayonier also found that drift within the bay was generally clockwise and easterly.¹

After the primary treatment system was installed at the mill, clarified and treated effluent was discharged to a submerged outfall located near the center of the mouth of the Port Angeles Harbor. The outfall consists of a submerged outfall line approximately 7,900 feet long that connects with an approximately 940-foot diffuser section containing 48 vertical outlet ports. Outfall location studies found that the currents rotated clockwise during receding or outgoing

tides, and moved toward the east during incoming or rising tides. Secondary treatment was added to the system in 1979. From the initiation of secondary treatment until operations ceased in 1997, the mill discharged approximately 50,000 pounds of suspended solids per day through the diffuser outfall.¹

The Tribe located other studies of current movement that support the earlier findings obtained by Rayonier. A pulp mill effluent study conducted in the early 1980s found that the effluent discharging from the submerged outfall was buoyant, warm, freshwater that migrated in an easterly direction towards Green Point and Dungeness Spit. During that same study, dye experiments indicated that portions of effluent released at the diffuser move to Morse Creek and Green Point.³ The Tribe also located drift card data, plume tracking data, and toxicity data that indicates that much of the effluent and emissions from the Rayonier mill site moved in an easterly direction, towards Dungeness Spit.² The National Oceanic and Atmospheric Administration investigated currents near the submerged Rayonier outfall by releasing 700 drift cards in the late 1970s. Of the 273 cards recovered, 65% were recovered between Ediz Hook and Dungeness Spit. Less than 10% moved to locations north or west of Ediz Hook.⁴ A drift card study conducted as part of a proposed pipeline route study found 73% of recovered cards released at the mouth of the Port Angeles Harbor were recovered between Ediz Hook and Dungeness Spit.⁵ While these data indicated that effluent migrated eastward, the data is not conclusive relative to contamination of biota. Actual testing is necessary to determine the level of contamination to the east of the mill site.

Recent effluent dispersion modeling conducted by Rayonier suggests that although there is an eastward component to the effluent, the suspended sediments tended to fall out near the outfall.² These modeling results were not verified by either the Tribe, or the Washington State Department of Ecology. Based on the results of Rayonier's dispersion modeling, the Rayonier RI sampling plan for the marine environment does not provide for sampling at a distance to the east of the mill site. Exclusion of samples in the Morse Creek to Dungeness Spit area from the RI leaves a data gap that is of concern to the Tribe.

Environmental Contamination

From the initiation of secondary treatment until operations ceased in 1997, the Rayonier Mill discharged approximately 50,000 pounds of suspended solids per day through its outfall. The primary contaminants of concern are dioxins and furans. These chemicals were measured in effluent at a concentration of approximately 4.9 mg TEQ/day, until the mill closed in 1997.

Toxicity tests on receiving water conducted in the area between the tip of Ediz Hook and Dungeness Spit in the 1960s and 1970s showed > 50% oyster toxicity (percent abnormal development) throughout the area. The toxicity was subsequently linked to pulp mill effluents in Port Angeles Harbor, and indicates that effluent, and associated chemicals, were transported to areas east of Port Angeles Harbor at concentrations sufficient to cause biological effects.³

There is no available tissue data to determine the Tribe's potential health risk from consuming Dungeness Crab (*Cancer Magister*) and geoduck (*Panope Abrupta*). Muscle tissue from a red rock crab (*Cancer Productus*) sample collected at Green Point in 1995 exceeded the 0.021 parts per trillion (ppt) EPA Region 3 Risk-Based Criteria for 2,3,7,8-TCDD.⁶ The dioxin concentration in the Green Point sample was 0.11 ppt TEQ, based only on detected congeners. This sample indicates the potential for marine organism uptake at concentrations that exceed EPA criteria.

The presence of effluent from the Rayonier mill in the Morse Creek/Dungeness Spit area, the concentrations of dioxins present in the effluent, the duration of effluent discharge, and the presence of dioxins in the Green Point tissue samples have raised concerns among the Lower Elwha Klallam Tribe about the safety of subsistence-level consumption of shellfish harvested from that area. The marine waters and associated resources between Port Angeles Harbor and Dungeness Spit are important to tribal members. The primary resource species of interest in this area are Dungeness crab and geoduck. Geoducks have a significant potential for contaminant bioaccumulation due to their long lifespan.

Discussion

This section (and the Appendices) describes the sampling and analysis tasks necessary to support the joint DOH/Tribe shellfish (Dungeness crab and geoduck) investigation, including the sampling approach and rationale for the number and location of samples to be collected. It also presents descriptions of sampling tasks, including descriptions of sampling equipment and collection methods to be used, the analyses to be performed, specifications for sample identification, a description of sample documentation, and identification of appropriate sample handling and management procedures. Sampling is anticipated to occur in October 2002. A separate health consultation will be prepared by DOH evaluating the results of the sampling analysis.

Sample Collection

Dungeness crabs

Dungeness crabs will be collected from five target sample areas previously identified by the Tribe. If Dungeness crabs are not available in sufficient numbers, red rock crabs may be collected as an alternative species, or adjustments may be made at the time and/or place of sampling. This determination will be made by field personnel in consultation with management personnel. The target areas are situated between Morse Creek and the base of Dungeness Spit (Figure 1). Five individual crabs will be collected from each target area, and combined, to represent a single, composite crab sample for that area. The five composite samples will represent a total of 25 crabs (five crabs per area for five areas). Given the limited resources, this sampling design provides the best combination of sample coverage and sample quantity. Multiple sampling areas and multiple individuals within each sample area will provide a reasonable representation of the overall population found in the study area. Individual crabs within each area will be collected from different sampling locations, perpendicular to the shoreline, to reduce any potential bias caused by sampling from different depths at different stations. Representative crabs collected from multiple depths will be selected to form the composite sample from each area. Individual locations within each area will be selected by the Tribe, while in the field. The Tribe anticipates sampling from individual locations between the shoreline, to at least one half mile offshore.

Dungeness crabs will be collected using standard commercial crab pots and ancillary gear. The pots will be fished from the Tribe's and/or others boats. Up to 10 pots may be used at each sample location. Sample collection equipment will be uncontaminated with grease or oil. The pots will be baited with fish, squid, clams, or similar material. The bait will be placed in small containers inside the trap to disperse the smell of the bait and attract the crabs.

Efforts will be made to harvest those crabs with a carapace length greater than 6.25 inches, as this is the current legal minimum size limit for Dungeness crabs. The preference will be for male crabs (the current legal catch). However, female crabs, and/or crabs smaller than 6.25 inches will be used if sufficient numbers of larger male crabs cannot be collected. The determination of

which crabs that will be retained for analysis, based upon size and sex, will be made by field personnel in consultation with management personnel. Individual crabs will be photographed, measured, weighed, and examined for any discoloration, abnormalities, or lesions. Normally, only unbroken crabs will be retained for analysis. Each crab will be killed by a rapid blow to the area on the underside of the carapace below the eyestalks, or by asphyxiation using dry icegenerated carbon dioxide. The crab will then be wrapped in decontaminated aluminum foil, bagged in a zip loc bag, labeled, and placed in a cooler with Blue Ice®, or similar product, not to be frozen. The coolers will be secured, labeled, and shipped with the necessary documentation and shipping papers after each day's sampling catch, by overnight delivery to AXYS Analytical, Inc. (AXYS Analytical) for sample processing and analysis. Samples will be shipped no later than Thursday of a given week. If samples cannot be shipped by Thursday, they will be held until the following Monday.

Geoducks

Geoducks will be collected from the same five target areas as the crabs, although specific locations within each area may be different, depending on where the geoducks are found. As with the crabs, each composite sample will consist of five individual geoducks from each of the five areas, for a total of 25 geoducks. Individual geoducks within each area will also be collected from different depths. Efforts will be made to collect geoducks representative of the sizes typically harvested and consumed by the Tribe. Geoducks will be collected by divers using hydraulic pressure excavation (gas-powered water pump or "dig pump" with a 3/4" diameter nozzle). Clams will be brought to the surface in a collection bag.

Individual geoducks will be measured, weighed, and examined for any discoloration, abnormalities, or lesions. The geoducks will be photographed, wrapped in decontaminated aluminum foil, placed in zip lock bags, labeled, and placed in a cooler with Blue Ice®, or similar product, not to be frozen. The coolers will be secured, labeled, and shipped with the necessary documentation and shipping papers after each day's sampling catch, by overnight delivery to AXYS Analytical for sample processing and analysis. Samples will be shipped no later than Thursday of a given week. If samples cannot be shipped by Thursday, they will be held until the following Monday.

Red rock crabs

If sufficient numbers of Red Rock crabs are available during the field sampling effort, red rock crabs may be collected at the same time and from the same five areas as the Dungeness crabs and geoducks to evaluate the correlation (if any) in dioxin levels. To the extent possible, sample collection, identification, shipping, preparation, and analysis will be identical to those used for the Dungeness crabs. Deviations from this plan may be made by field personnel in consultation with management personnel.

Reference area samples

Reference samples will be collected from areas east and west of the study area that are not likely to be significantly affected by releases from the former Rayonier Mill. Since low levels of dioxins and furans are ubiquitous in the environment (including in shellfish), it is useful to compare levels in shellfish that reside in areas likely to have been impacted by industrial sources, such as the former mill site, to levels in shellfish in areas relatively unimpacted by industrial sources (i.e., reference areas). For this exposure investigation, Dungeness Spit and Freshwater Bay are the selected reference locations. Dungeness Bay is a protected, nonurbanized area approximately 15 miles east of Port Angeles Harbor. Freshwater Bay is a semi-protected bay located approximately 10 miles west of Port Angeles (Figure 1).

Since Rayonier is also using Dungeness Spit and Freshwater Bay as reference areas for their remedial investigation (RI), they agreed to provide the Tribe with splits of their Dungeness crab and geoduck samples from both reference areas for the DOH/Tribe exposure investigation. This will provide the necessary reference samples with which to compare the non-reference area shellfish samples collected during the exposure investigation.

Six of Rayonier's split reference shellfish samples will be sent overnight from Columbia Analytical in Houston (where Rayonier's dioxin/furan analysis is being conducted) to AXYS Analytical for analysis. The six split samples include one individual geoduck sample from each reference area, and two composite crab samples from each reference area. The composited crab samples will consist of one composite muscle tissue sample, and one composite crab "butter" sample (which includes the hepatopancreas tissue) from each reference area. Each crab sample will be a composite of five to eight individuals, depending on the success of the Rayonier sampling effort in obtaining sufficient individuals. Columbia Analytical will ship the composited and homogenized samples directly to AXYS Analytical.

The dioxin/furan analysis requires a minimum of 10-12 grams of shellfish tissue for each sample composite. Rayonier has indicated that Columbia Analytical should have this quantity remaining for analysis by AXYS after completion of their own analysis. If this minimum quantity of shellfish homogenate is not available from Columbia, reference samples will be collected by the Tribe in the same manner as the other samples. If this occurs, the number of non reference samples analyzed may be reduced due to budget constraints.

Laboratory processing of shellfish samples

As described above, AXYS Analytical will analyze the six split samples provided by Rayonier's lab. In the unlikely event AXYS Analytical determines that additional homogenization of Rayonier's split shellfish homogenate is necessary, they will notify DOH and/or the tribe prior to analysis. If any other problems are noted (i.e., temperature abnormalities, condition of samples, condition of container, etc.), DOH and the Tribe will be notified.

All shellfish samples collected by the Tribe during the EI will be processed and analyzed by AXYS Analytical according to the procedures outlined in this sampling workplan and as described in the U.S. Environmental Protection Agency Guidance for Assessing Chemical Contaminant Data for Use In Fish Advisories. Processing includes sample-logging and inspection, removal of edible portions of the shellfish, compositing, weighing, homogenizing, aliquot preparation, etc. Special instruction for processing or analysis, if any, will be noted by DOH on the analytical request form.

Crabs

Rayonier's split samples: Columbia Analytical will send, (by overnight delivery) to AXYS Analytical, pre-composited and pre-homogenized crab muscle tissue (two) and crab butter (two) samples. No additional processing is anticipated for these split reference samples. AXYS Analytical will evaluate the samples for sample integrity, ensure they are sufficiently homogenized, weigh them, and analyze the samples for lipids, and for dioxins/furans by EPA method 1613B.

DOH/Tribe EI samples: Whole crabs collected and submitted during the DOH/Tribe exposure investigation will be evaluated for sample integrity. Each individual will be measured and weighed prior to dissection and homogenization, and its sex noted. Since tribal members often consume the crab butter and crab muscle portions together, AXYS Analytical will combine and homogenize the crab butter and crab muscle portions of each sample composite collected by the Tribe. Shellfish tissue splits obtained from Columbia Analytical will not be recomposited by AXYS Analytical.

Edible portions of the muscle tissue (legs and body) and crab butter from the five individual crabs that make up each composite will be removed from the shell, combined, and weighed. Each composite sample (consisting of crab butter and muscle tissue from five crabs) will then be homogenized, for a total of five separate composite crab samples. Aliquots of the samples will be prepared and analyzed by EPA method 1613B for dioxins and furans. In addition, a lipids analysis will be conducted for each sample.

Geoducks

Rayonier's split samples: As discussed above, two individual geoduck samples from the reference areas will arrive from Columbia Analytical pre-weighed and homogenized. AXYS Analytical will evaluate the integrity of the geoduck homogenate. If no additional homogenization is necessary, the samples will be analyzed for lipids, and for dioxins/furans by EPA method 1613B.

DOH/Tribe EI samples: AXYS Analytical will evaluate the integrity of the samples, and weigh the geoducks. Edible portions of the geoduck muscle tissue (neck) will be separated from the shell and gutball prior to homogenization. The five individual geoduck samples representing each

sampling area will be composited into a single sample, homogenized, then analyzed, for a total of five composite samples (one composite representing each area). The samples will be analyzed for lipids, and for dioxins/furans by EPA method 1613B.

Data reporting and evaluation

Upon completion of the sample analysis, AXYS Analytical will prepare data summary reports. The reports will include the results of the chemical and lipid analysis, and the appropriate quality assurance and quality control reports as required by EPA method 1613, including initial calibration, calibration verification, GC column resolution, procedural blank and ongoing precision and recovery results. A full data package will also be provided which will include a narrative discussion, example calculations, sample receiving records, sample homogenization records, wet chemistry logs and all raw data including window defining results, injection logs and test sample and QA sample chromatograms.

The results of the analysis will be evaluated by DOH in a health consultation that, upon completion, will be available to the Tribe, Rayonier, Ecology, and the public. Since the split crab butter and crab muscle reference samples obtained from Rayonier will be analyzed separately by AXYS Analytical (as opposed to the combined crab muscle and crab butter analysis conducted for the EI shellfish samples), the sample mass will be used to compare and evaluate the results of the reference and non-reference crab samples.

Conclusions

- 1. The Lower Elwha Klallam tribe collects and consumes Dungeness crabs, geoducks, and other aquatic resources from the marine environment in the vicinity of the Port Angeles Harbor where dioxin and furan-containing effluent may have migrated.
- 2. No conclusive data currently exist to determine whether dioxin/furan levels in Dungeness crabs and geoducks in the marine environment in the vicinity of the Port Angeles Harbor pose a health threat to the tribe.
- 3. This data gap can be addressed by the collection and analysis of Dungeness crabs and geoducks in the target areas identified in the Discussion section.

Recommendations/Action Plan

- 1. Dungeness crab and geoduck sampling, preparation, and analysis should be conducted according to the methods outlined in this exposure investigation workplan.
- 2. DOH will evaluate the results of the sampling analysis to determine whether the detected levels of dioxins and furans pose a health threat to members of the Lower Elwha Klallam tribe who harvest and consume Dungeness crabs and geoducks from the target sample areas.
- 3. The results of DOH's evaluation will be provided to the Tribe, Ecology, Rayonier, and the community. Possible recommendations include additional sampling, limits on the amount of crabs and geoducks consumed by the Tribe, or no further action.

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- 9. Washington State Department of Health. Draft Exposure Investigation Protocol: Marrowstone Island, Washington. April 1998.
- 10. Personal communications with Laurie Phillips, AXYS Analytical Services, Ltd. July/August 2002.
- 11. Personal communications with Bruce Woods, analytical chemist, United States Environmental Protection Agency. July 2002.
- 12. Personal communication with Roy Hummell, Project Manager, Foster Wheeler Environmental Corporation. July/August 2002.

Appendices

Following is a description of sampling materials, navigation and positioning, field sampling methods, documentation, sample packing and shipping, laboratory analytical methods, quality assurance, data reporting, and data analysis.

Materials:

The materials needed for sampling and shipping should be verified with AXYS Analytical (the contract laboratory that will conduct the analysis). The following list of materials is anticipated:

Boat (provided by the Tribe)

Cotton gloves

Latex gloves

Chain-of-custody forms

Chain-of-custody sealing tape

Sampling log forms

Rite-in-the Rain® paper

Global positioning system (GPS) unit

Ice chests

Blue Ice® (or equivalent)

Packing material

Labels

Aluminum foil (2 packages, minimum)

Plastic ziplock bags.

Plastic wrap or bags (rain protection for forms)

Black markers with waterproof ink

Pens

Digital camera (substitute disposable camera in foul weather)

Crab pots, crab bait, and appurtenant gear

Tape measure

Crab measure

New, clean, decontaminated spray can (one gallon size)

Distilled water for final rinse of decontaminated equipment

Geoduck collection equipment (geoduck harvest pump or "dig" pump) per standard tribal harvesting practices

Navigation and positioning:

Vessel navigation and positioning during the EI sampling effort will consist of field determinations of sample locations based on the information contained in the discussion above and the attached figures, with particular emphasis on the depths of the samples. The locations of the sample transects were chosen to match prominent geographical features to aid in positioning the vessel during sampling. Actual sample locations will then be noted by use of Global Positioning System (GPS) equipment. The objective of the sample location positioning procedure is to accurately identify and record the location of all sampling locations. The GPS will generate positions (latitude/longitude), to at least 10 meters of accuracy.

Field Sampling Methods:

The following steps will be used to collect, document, and process the shellfish samples:

- 1. Collect marine biota samples using conventional sampling techniques and equipment, as described in the discussion section.
- 2. Using clean, unused, disposable, un-powdered gloves, place sample in decontaminated aluminum foil (dull side in) and place in a plastic Ziploc ® bag, along with a sample ID tag.
- 3. Cool samples to a temperature less than 4° C by placing in an insulated cooler and surrounding with Blue Ice® packs, or similar product. Do not freeze the samples. Protect samples from light.
- 4. Label and manage sample containers. Labels should include the project name, site and sample number (i.e., Pt. Angeles Exposure Investigation, Site 1, Location 2). Sample collection information should be entered on a sample collection data form, which includes sufficient information for future determination of GPS data. Finally, a chain of custody form should be filled out and placed in a large ziplock bag inside the chest sealed with either chain of custody tape or other device. Iced samples should be sent to the designated laboratory by overnight delivery, or as soon thereafter as possible, as specified by the contracted laboratory for initial sample preparation.
- Decontaminate sampling equipment in accordance with EPA Guidance for Assessing Chemical Contaminant Data for Use In Fish Advisories, if such equipment will be reused.

Sources of extraneous tissue contamination include contamination from sampling gear, grease from ship winches or cables, spilled engine fuel (gasoline or diesel), engine exhaust, dust, ice chests, and Blue Ice® used for cooling. All potential sources of contamination in the field should be identified and appropriate steps taken to minimize or

eliminate them. For example, during sampling, the boat should be positioned so that engine exhausts do not fall on the deck. Ice chests should be scrubbed clean with detergent and rinsed with distilled water, and sealed before being taken on the boat. To avoid contamination, samples should be handled with new, clean, disposable gloves only, after being taken aboard the boat. When not being handled, samples must only be placed on the dull side of unused foil or kept in life tanks with clean, fresh seawater. After field measurements, tagging, and wrapping in foil, the samples shall placed in waterproof plastic bags. Sampling equipment that has obviously been contaminated by oils, grease, diesel fuel, or gasoline should not be used. All utensils or equipment that will be used directly in handling shellfish (e.g., shellfish measuring board or calipers) should be cleaned prior to each sampling trip, rinsed in acetone and pesticide-grade hexane or Alconox, distilled water decon, and stored in aluminum foil until use. Between sampling sites, the field collection team should clean each measurement device by rinsing it with ambient water, followed by a rinse with distilled water, and rewrapping it in aluminum foil to prevent contamination.

6. Marine biota (shellfish) samples collected as part of the EI will be composited in the laboratory.

Documentation:

Photographs will be taken throughout the field sampling effort to document samples. In addition, field sampling information should be recorded with, at a minimum, the following information for each target sampling area:

| AREA # | GPS Coordinates | Location Description | Species - Number Collected | Notes | Date |
|--------|-----------------|----------------------|-------------------------------|-------|------|
| | Sub-Area 1 | | | | |
| | Sub-Area 2 | | | | |
| 1 | Sub-Area 3 | | | | |
| | Sub-Area 4 | | | | |
| | Sub-Area 5 | | | | |

Sample packing and shipping

All samples collected will be assigned unique sample numbers, labeled, noted in waterproof ink in a bound, Rite-in-the-Rain® field logbook, and recorded on the chain-of-custody form. Labels for sample containers will be filled out completely with all appropriate information. Samples will then be packed for shipment to the laboratory according to the current U.S. Department of Transportation (DOT) and Washington Administrative Code (WAC) 173-303-071(3)(1) requirements. Sample containers will be packed in coolers with a low-density packing material, and Blue Ice® (or equivalent). The coolers will be securely sealed.

All samples will be shipped via express delivery for overnight delivery to the contracted laboratory (AXYS Analytical). A commercial invoice supplied by AXYS Analytical will be used to facilitate delivery of the containers across the U.S. - Canadian border. Custody seals will be used on coolers. Samples will be shipped no later than Thursday of a given week. If samples cannot be shipped by Thursday, they will be held on ice until the following Monday. A minimum of 10 bags of Blue Ice® per chest will be necessary to hold temperature for as long as two days in the event samples are delayed in shipment.

Upon receipt at the laboratory, the custody seal will be broken, and the receiver will record the condition of the samples. The chain-of-custody form will be signed. The samples will be logged into the Laboratory Information Management System (LIMS) to allow the samples to be tracked while in the custody of the Laboratory. Sample security will be assured since the AXYS Analytical premises are locked with restricted access at all times. After homogenization, the samples will be maintained frozen until analyses are completed. Final disposition of any remaining sample will be decided in consultation with the DOH and the Tribe.

Laboratory Analytical Methods

AXYS Analytical Services (Sydney, B.C.) will analyze shellfish samples received from both Columbia Analytical (Rayonier's lab), and from the joint DOH/tribal exposure investigation. Samples received from Columbia Analytical will be pre-homogenized. Shellfish samples from the EI will be sent whole. The sample analysis will be performed by EPA method 1613B, using high resolution GC/MS. The method includes analysis of 17 dioxin and furan congeners. Method detection limits are expected to be in the range of 0.02 per trillion (ppt) to 1 ppt, depending on the congener and sample size available for analysis. Lipid analysis will also be performed on each sample.

Quality Assurance/Quality Control

Split shellfish tissue samples will be obtained from Rayonier, to be analyzed separately by AXYS Analytical. The results will be included with the analysis report for the EI samples. Standard laboratory quality control procedures will be utilized, including method and instrument blanks, surrogate recoveries, and laboratory control samples. In addition AXYS Analytical will include a sixth initial calibration point (5 times lower than the method stated CS-1) to allow reporting uncensored data to the lowest level possible. All method QA/QC requirements will be met for instrument tuning, resolution and calibration prior to sample analysis.

Data Reporting

AXYS Analytical will prepare data analysis reports within two weeks of completing the analysis, summarizing the results of all samples (field, split, and lab QA). The reports will include the results of the chemical and lipid analysis, and all quality assurance and quality control reports as required by EPA Method 1613 including initial calibration, calibration verification, GC column resolution, procedural blank and ongoing precision and recovery (OPR) results.

Data Analysis

AXYS Analytical will provide a data analysis package upon completion of the project. The data will be evaluated by DOH in a health consultation, which will be provided to the Tribe, Ecology, Rayonier, and the community.

Certification

| cooperative agreemer | tion was prepared by the Washington State Department of Health under a at with the Agency for Toxic Substances and Disease Registry (ATSDR). It approved methodology and procedures existing at the time the health un. |
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| | Debra Gable |
| | Technical Project Officer, SPS, SSAB, DHAC |
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| | th Assessment and Consultation, ATSDR, has reviewed this public health curs with the findings. |
| consultation and conc | urs with the findings. |
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| | Roberta Ehrlwein |
| | Chief, SPS, SSAB, DHAC ATSDR |
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